(4)

CHEMICAL
RESEARCH,
DEVELOPMENT &
ENGINEERING
CENTER

CRDEC-CR-067

AD-A223 558

SUBMICRON PARTICLE ANALYZER

Philip J. Wyatt, Ph.D.
Christian Jackson

WYATT TECHNOLOGY CORPORATION
Santa Barbara, CA 93103-3003

April 1990

U.S. ARMY
ARMAMENT
MUNITIONS
CHEMICAL COMMAND

Aberdeen Proving Ground, Maryland 21010-5423

Disclaimer

Distribution Statement

UNCLASSIFIED
SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

1a. REPORT SECURITY CLASSIFICATION
UNCLASSIFIED

1b. RESTRICTIVE MARKINGS

2a. SECURITY CLASSIFICATION AUTHORITY

2b. DECLASSIFICATION / DOWNGRADING SCHEDULE

3. DISTRIBUTION / AVAILABILITY OF REPORT
Approved for public release; distribution is unlimited.

4. PERFORMING ORGANIZATION REPORT NUMBER(S)
CRDEC-CR-067

5. MONITORING ORGANIZATION REPORT NUMBER(S)

6a. NAME OF PERFORMING ORGANIZATION
Wyatt Technology Corporation

6b. OFFICE SYMBOL *(If applicable)*

7a. NAME OF MONITORING ORGANIZATION

6c. ADDRESS *(City, State, and ZIP Code)*
P.O. Box 3003
Santa Barbara, CA 93103-3003

7b. ADDRESS *(City, State, and ZIP Code)*

8a. NAME OF FUNDING / SPONSORING ORGANIZATION
CRDEC

8b. OFFICE SYMBOL *(If applicable)*
SMCCR-RSP-B

9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER
DAAA15-85-C-006

8c. ADDRESS *(City, State, and ZIP Code)*
Aberdeen Proving Ground, MD 21010-5423

10. SOURCE OF FUNDING NUMBERS

PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT ACCESSION NO.

11. TITLE *(Include Security Classification)*
Submicron Particle Analyzer

12. PERSONAL AUTHOR(S)
Wyatt, Philip J., Ph.D., and Jackson, Christian

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15. PAGE COUNT
Contractor	FROM 85 May TO 87 Dec	1990 April	33

16. SUPPLEMENTARY NOTATION
COR: Jerold Bottiger, SMCCR-RSP-B, (301) 671-2395

17. COSATI CODES

FIELD	GROUP	SUB-GROUP
20	06	

18. SUBJECT TERMS *(Continue on reverse if necessary and identify by block number)*
Aerosol; Particle analyzer; Light scattering; Aerosol identification; Obscurants; Multiangle scattering

19. ABSTRACT *(Continue on reverse if necessary and identify by block number)*

Two submicron aerosol particle analyzers of increasingly complex design were developed, under a Phase II SBIR grant. The final objective of this program was the development of a real-time aerosol characterization and identification system for particles in the 100 to 1500 nanometer size range. Two completed systems were delivered to the Army contractor. Each system was able to collect the light scattered from individual aerosol particles at, respectively, 16 and 24 angles. Data at all angles can be collected at rates in excess of 200 particles per second. Major breakthroughs were made in the design and implementation of real-time data-processing hardware, but due to budget limitations, this incomplete software aspect of the system was not incorporated into the final design.

20. DISTRIBUTION / AVAILABILITY OF ABSTRACT
☒ UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT. ☐ DTIC USERS

21. ABSTRACT SECURITY CLASSIFICATION
UNCLASSIFIED

22a. NAME OF RESPONSIBLE INDIVIDUAL	22b. TELEPHONE (Include Area Code)	22c. OFFICE SYMBOL
SANDRA J. JOHNSON	(301) 671-2914	SMCCR-SPS-T

DD Form 1473, JUN 86 *Previous editions are obsolete.* SECURITY CLASSIFICATION OF THIS PAGE

UNCLASSIFIED

PREFACE

The work described in this report was authorized under Contract No. DAAA15-85-C-006. This work was started in May 1985 and completed in December 1987.

The use of trade names or manufacturers' names in this report does not constitute an official endorsement of any commercial products. This report may not be cited for purposes of advertisement.

Acknowledgments

The authors wish to acknowledge the contributions of Steven D. Phillips, Dr. David T. Phillips, Dr. Jerold R. Bottiger, David L. Hicks, Yu-Jain Chang, and Dr. Kevin L. Schehrer.

Blank

CONTENTS

LIST OF FIGURES

SUBMICRON PARTICLE ANALYZER

1. INTRODUCTION

Two Submicron Aerosol Particle Analyzers were developed, fabricated, and tested under a Phase II Small Business Innovation Research (SBIR) grant. The Phase I program (DAAA15-85-C-006) confirmed feasibility of the concept of using multiangle scattering data for the rapid characterization/identification of single particles. A preliminary design was established and some electronic circuitry tested[1]. Under Congressional guidelines, the Phase II program of an SBIR program has as its primary objectives the completion of preproduction engineering and preparation for the commercialization of the systems developed. These objectives have been achieved. The instruments were designed to characterize aerosols by the measurement of simultaneous, multi-angle light scattering observations from single aerosol particles passing perpendicularly through a laser beam.

Both systems center around a precision machined (100 mm o.d. 40 mm i.d.) spherical scattering chamber. The laser beam from a He-Cd laser lies along a diameter of the sphere and intersects a particle stream passing perpendicular to it at the center of the chamber. The surface of the sphere contains 72 collimated detector ports arranged on 4 great circles having the laser beam as a common diameter. Each circle has 18 ports and all are oriented radially, i.e. perpendicular to the surface of the sphere. There are also two large fiber bundle ports subtending an azimuthal angle about the laser beam of nearly 2π and centered about the polar angles 25° and 155° with respect to the laser beam direction of propagation. Optical fibers with collimating lenses attached to their faces fit interchangeably into any of the 72 ports. These fibers and the fiber bundles terminate at the photocathodes of an array of photomultiplier tubes (PMTs). Signals collected by the fibers and transmitted to the PMTs are amplified and transmitted to the particle detection, peak location, and sample-and-hold circuitry. All electronics are under software control from an IBM XT/AT compatible computer. An aerosol handling system which controls aerosol particle concentration and speed includes sampling aperture, pumps, filters, and valves .

The first system delivered to the Obscuration and Aerosol Sciences Laboratory included 14 optical fibers with collimating lenses and two fiber bundles. Scattered light was collected, therefore, from 16 angular positions on the scattering sphere. These signals were amplified first by photomultiplier tube detectors and subsequently by logarithmic amplifiers to handle the large range of expected signals (of the order of 5 orders of magnitude). Peak detection and sample-and-hold circuitry permitted the simultaneous capture at all 16 angles of the scattered spherical wavefront signal produced by each particle during its traversal of the laser beam. These signals were then stored in an IBM XT compatible computer memory using a 16 channel analog-to-digital board whose maximum conversion rate is 27.5K channels (signals) per second.

In the second instrument, we had hoped to replace the PMTs by more rugged and compact solid state photo-detectors. MAMA tubes, image intensifiers and avalanche photodiodes were all extensively investigated but for reasons discussed in Sec. 2.3, PMTs were found to be superior, in terms of sensitivity, dynamic range, and cost. The second system computer selected was a powerful IBM AT compatible, and a much faster A/D board, with a maximum conversion rate of 112K channels/sec., was used. The number of scattering angle data was expanded to 24 corresponding to 22 optical fibers and 2 fiber bundles. The improved computing power enabled us to start work on real-time particle characterization using a Definicon card. Experimental results from the first instrument had revealed that for low signals the logarithmic amplifier response time was far slower than expected. A new dual serial linear amplifier system was designed for each detector and integrated into the system. This enabled us to measure the full range of expected signals with greatly improved speed and precision. Furthermore, experience in operating the first instrument enabled us to locate problem areas in that design and to develop solutions. Most notably, the sealing of the scattering chamber was improved by changes in the input and output port structures. In addition the laser alignment mechanism was made both simpler and more precise. Data collection software was also enhanced.

The final design of this second instrument is discussed below. A description of the first instrument, along with some typical data, has been published recently[2].

2. THE INSTRUMENT

The Submicron Particle Analyzer system allows the light scattered from individual particles as they traverse a He-Cd laser beam to be measured and stored from 22 of 72 well defined possible positions on the surface of a spherical scattering chamber. In addition, light is collected at one forward and one rear large solid angle region. All 24 intensities are processed at rates of up to 200 particles per second. Some real-time analyses of the collected data are easily performed (e.g. symmetry and depolarization phenomena, estimates of particle size, etc.). More detailed analyses may be performed after collection.

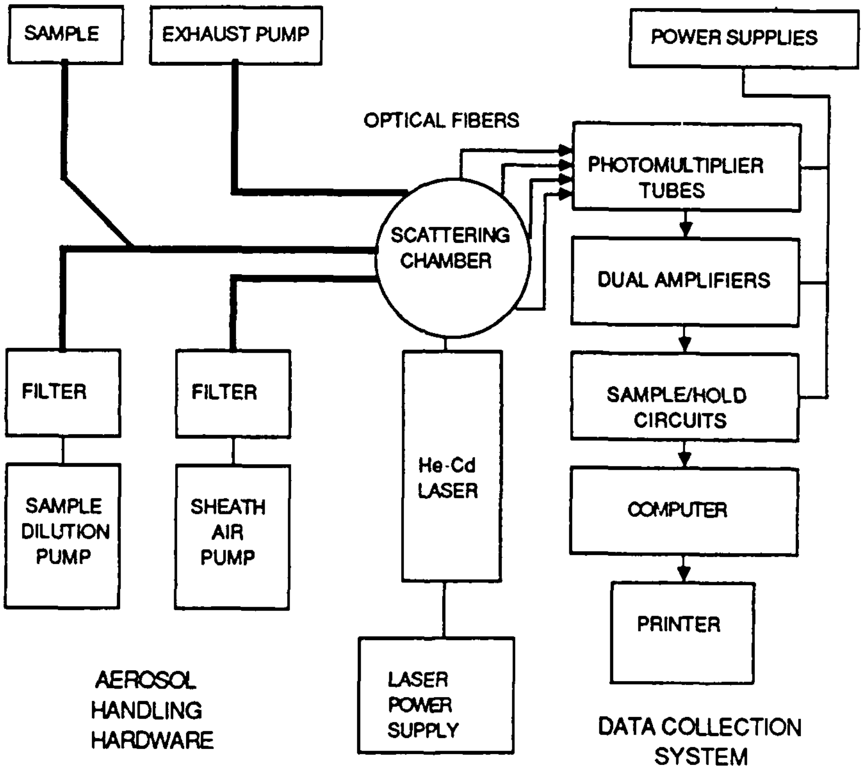

Figure 1. The Main Components of the Aerosol System

The aerosol system consists of six main components (see Fig.1), (1) a He-Cd laser light source, (2) a high-precision spherical scattering chamber, (3) 24 scattered light detectors consisting of 22 collimating lenses and optical fibers, two fiber bundles, and 24 photomultiplier tubes, (4) particle detection and data collection electronic circuitry, (5) data storage and processing software running on a PC-AT, and (6) aerosol handling hardware to sample, dilute and introduce particles into the scattering chamber. The photomultiplier tubes, electronics, aerosol handling system and all power supplies are housed together (Fig. 2).

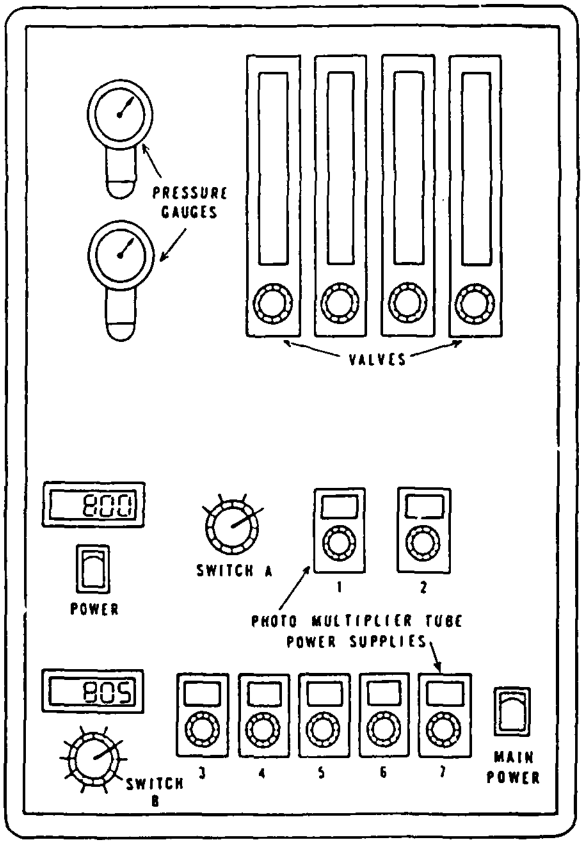

Figure 2. Aerosol System Front Panel

2.1 *Laser.*

The laser light source used is a 10 mW He-Cd laser emitting a 442 nm plane polarized beam of nominal diameter 0.3 mm. This beam is aligned through the center of the scattering chamber, along the common axis of the four great circles of the detector ports and perpendicular to the particle flow (see Figs. 3 and 4), by means of the beam steering mounts. These mounts allow precise alignment of the beam to be achieved and the self-locking mechanism ensures that it remains fixed in space.

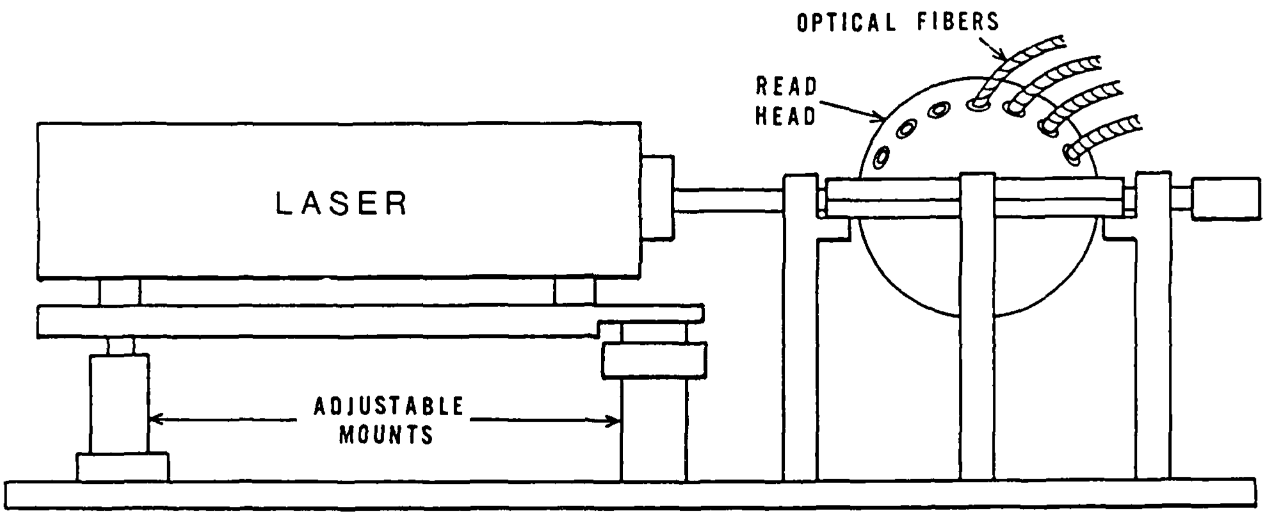

Figure 3. Optical Bench with Laser and Scattering Chamber

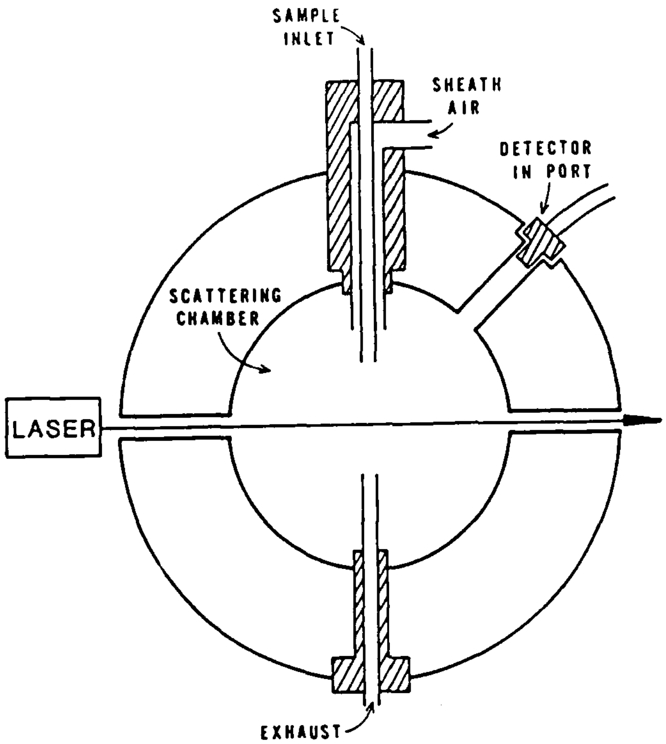

Figure 4. Scattering Chamber

The He-Cd laser was chosen to improve submicron particle detection; its short wavelength and narrow beam-width giving greater power density. The seals around the inputs and outputs of the scattering chamber were radically improved in this second instrument by re-machining the surfaces and using new gaskets. With the scattering chamber vacuum sealed, following these improvements, it is possible to control particle flow precisely. Where the signal size is too small it is now feasible to draw the particles through the beam with decreased velocity increasing the 'dwell' time in the beam and thus the signal resolution.

The laser manual was delivered with the instrument. The specifications follow:

Power	10 mW
Mode	TEM_{00}
Wavelength	442 nm
Beam diameter ($1/e^2$)	0.3 mm
Beam divergence	1.9 μradians
Polarization	> 500:1

2.2 *Scattering Chamber.*

The scattering chamber is a sphere, comprised of two hemispheres, with an internal diameter of 40 mm and outer diameter of 100 mm. There are entry and exit apertures for the laser beam and the particle stream (which are at right angles to each other).The 74 detector positions consist of two large ports for fiber bundles, subtending an azimuthal angle of almost 2π and about $10°$ of polar angle centered on $25°$ and $155°$, respectively. The remaining 72 small ports lie on four great circles. These great circles have the laser beam as a common diameter, and are arranged at $45°$ intervals, the first being parallel to the plane of polarization of the incident laser beam (Fig. 5). These ports are either fitted with a removeable sealing fixture or an optical collimator. The plane of polarization of the incident laser beam is adjusted to lie at $22.5°$ from the vertical by means of a quarter-wave plate. This is so that the detector ports can be placed in the plane parallel to the plane of polarization without interfering with the particle input and exhaust structures which are placed between the great circles.

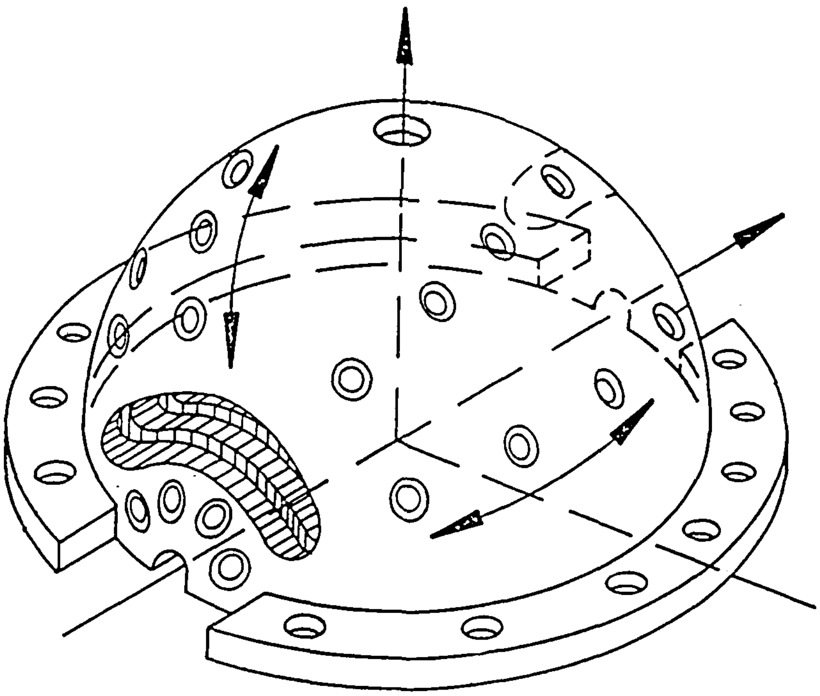

Figure 5. Detector Port Positions

2.3 *Scattered light detectors*

For insertion into any of the 72 small ports, each of the 22 detector structures consists of an optical collimator with an acceptance angle of ± 1.25° attached to the photocathode of a photomultiplier tube by 1 meter of optical fiber. An optical collimator is comprised of a 1.7 mm half pitch gradient refractive index (GRIN) lens of numerical aperture 0.28, cemented to 140 μm optical fiber. We built and delivered special analyzers to be placed in front of the collimators in order to measure the polarized or depolarized components of the scattered light. These analyzers consisted of a small disk of polaroid attached to a fitting which could be placed in front of the optical collimators. This fitting could then be inserted into the scattering chamber port. The optical bundles collect light directly through two near-semicircular ends, which are polished and embedded in the scattering head. The two ends from each fiber bundle, one in the lower and one in the upper hemisphere of the scattering chamber, are coupled together and the coupled end is attached to the photocathode of a photomultiplier tube by means of index matching fluid.

Hamamatsu R647-04 and R1104HA photomultiplier tubes were used as detectors for the optical collimators and fiber bundles, respectively. Their specifications follow:

R647-04:
Designed for photon counting, HA coating with magnetic shield.
Spectral response: 300-650 nm, peak wavelength 420 nm
Photocathode material: Bialkali
Window material: Borosilicate glass
Dynode structure: Linear focused
Number of dynode stages: 10
Maximum anode to cathode voltage: 1250 Vdc
Maximum average anode current: 0.1 mA
Anode dark current: 5 nA typical
Cathode sensitivity: luminous 90 μA/lm typical
radiant 80 mA/W typical
Anode to cathode supply voltage 1000 Vdc for anode characteristics listed below
Anode sensitivity: luminous 200 A/lm typical
radiant 1.8×10^5 A/W typical
Current amplification: 2.2×10^6
Time response: electron transit time 24 ns typical

R1104HA:
Wide spectral response, multialkali photocathode; high gain
Spectral response: 185-850 nm, peak wavelength 420 nm
Photocathode material: Multialkali
Window material: UV glass
Dynode structure: linear focused
Number of dynode stages: 11
Maximum anode to cathode voltage: 1500
Maximum anode current: 0.1 mA
Anode dark current: 25 nA typical

Cathode sensitivity: luminous: 140 μA/lm typical
radiant: 60 mA/W typical
Anode to cathode supply voltage: 1000 Vdc for anode characteristics listed below
Anode sensitivity: luminous: 2000 A/lm typical
radiant: 8.4×10^5 A/W typical
Current amplification: 1.4×10^7
Time response: electron transit time 60 ns typical

One of our aims in Phase II was to find a photodetection system that at least would equal the sensitivity of the PMTs, but which would be much smaller, and thus more suitable for a portable field system.

In considering what type of photodetector might be appropriate for the Aerosol system, we calculated the approximate range of optical power incident on a detector as follows: assume a laser power of 10 milliwatts and beam diameter of 0.5 millimeters. The cross-sectional diameter of particles measured can range from about 100 nm to 1000 nm, giving a range of incident power from 4×10^{-10} W to 4×10^{-8} W. Let us assume isotropic scattering, and a 2° by 2° detector solid angle; then a detector sees about .015 of total scattering, or from 6×10^{-12} W to 6×10^{-10} W.

2.3.1 *Image intensifiers* .

These devices convert an incident light signal into a magnified outgoing signal, based on the physical principles of the photoelectric effect and the cathodoluminescent effect. The incident light strikes a semitransparent photocathode, which emits photoelectrons. The resulting electron image is amplified by a microchannel plate (MCP). A MCP consists of an array of glass capillaries, or channels, typically 15 microns in diameter, fused into a thin plate. The channels are coated with a material which emits secondary electrons when struck by electrons. After being accelerated by an electric field, the photoelectrons enter the MCP and are multiplied by a factor of about 1000. After further acceleration by an electric field, the photoelectrons strike the fluorescent screen, resulting in an optical output. The optical gain of MCP image intensifiers should be about 10,000.

Litton provided us with several image intensifiers, which we examined to determine their suitability as a relatively inexpensive way of amplifying the light signals obtained from the Aerosol scattering chamber. We attempted to couple the incoming light signal, via optical fibers, to the input of the image intensifier. The output was coupled to PMTs via additional optical fibers.

We found that good coupling was very difficult to obtain. The highest observed gain was about 1000. The image intensifier proved to have a limited dynamic range for our purpose. A "blooming" effect occurred when the incoming light exceeded a relatively modest level compared to the highest expected input. That is, if the input at a particular region of the photocathode were a circle of light emanating from a particular optical fiber, then the corresponding circular image at the fluorescent screen became much larger than the input circle of light, as the incident intensity increased. In the time available to us, we were unable to achieve a repeatable linear response to the range of incident optical power we calculated would occur during normal experiments.

2.3.2 *Multianode microchannel arrays (MAMAs)*

These are Micro Channel Plate (MCP) devices which convert incident light to an electrical output. They were designed for use in space astrophysics instruments. During the current phase, the long delivery time and high cost of these devices prevented us from purchasing one for testing.

The device is linear up to about 100,000 counts per second per anode. Since the maximum collection time corresponding to a single particle is about 100 μs, the maximum count would be about 10, a value incompatible with the broad dynamic range required.

2.3.3 *Avalanche photodiodes (APDs)*

These are silicon photodiodes with an extra gain provided by the avalanche effect. When the photodiode is operated under very high reverse bias, carriers traversing the depletion layer have enough energy so that when they collide with electrons in the valence band, these secondary electronics can be raised to the conduction band. Thus a new electron-hole pair is created. The secondary electrons may themselves cause the creation of further electron-hole pairs through collisions. The continual repetition of this process is the avalanche effect.

APDs are considered suitable for amplification of very low light levels. They have an ultra-fast response time and have been used successfully for photon counting. Therefore, it would seem they may be a suitable device for the Aerosol instrument.

One precaution which must be taken is the use of a very stable power supply, since gain is strongly dependent on the bias voltage. The gain is also strongly dependent on temperature. However, techniques have been developed to use APDs while taking these difficulties into account. For example, an identical control diode can be used to monitor fluctuations in gain and thus correct the detector diode.

Typically, there is a current gain of about 10 for a reverse bias voltage of 100 V. A reverse bias voltage of several hundred volts results in a current gain of about 100.

We were fortunate to have the cooperation of RCA Canada in examining the suitability of APDs for our work. They performed some simple tests of their C30921S APD under parameters designed to simulate light scattering in our Aerosol chamber. The result was promising and APDs seemed to be the best alternative to PMTs; however, we were unable to obtain samples for our own use in the time available to us, and we decided to postpone our consideration of APDs.

The RCA test involved light pulses of 50 microseconds, with a minimum power of 4.8×10^{-12} W, corresponding to about 1000 photons. The maximum power input corresponded to about 1,000,000 photons. The APDs themselves are reported to be linear over this range. Care must be taken to ensure that non-linearity is not introduced by the amplification circuit. The RCA test light source was an LED emitting at 830 nm. The resulting signal clearly showed that a particle generating this amount of scattering at a detector could be detected without difficulty. (See Fig. 6) . The device was operated in the linear mode; however, a photon counting mode is also available.

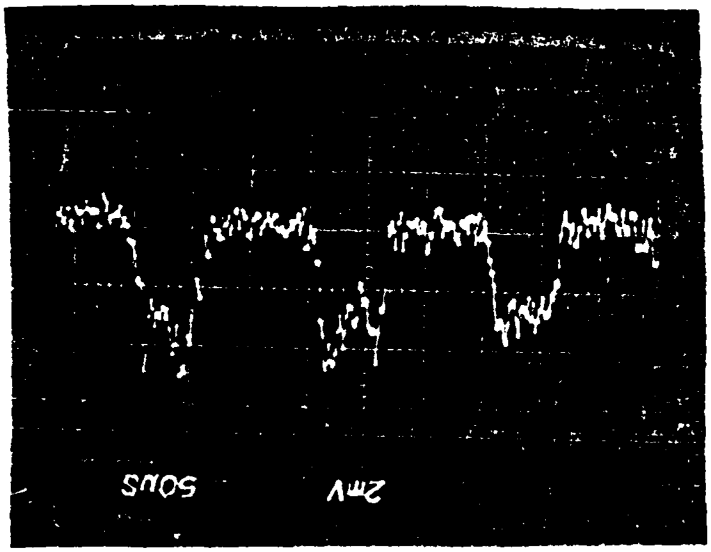

Figure 6. APD output signal for a pulsing LED

2.3.4 *Multi-anode PMTs.*

Hamamatsu makes a multi-anode PMT, the R2486, which, had it worked, would have allowed us a PMT-type response on several channels, but using a single tube. We believed this might be an alternative to the use of one PMT per channel, providing some degree of miniaturization, along with the proven capabilities of the PMT. However, Hamamatsu was unable to guarantee the desired maximum cross talk between channels, which we specified as 1%. Clearly, we do not wish to add any further sources of noise to our data channels.

2.3.5 *Miniature PMTs.*

Hamamatsu makes a 10 mm diameter PMT, the R1635, with specifications similar to those of the R647 except that the anode characteristics similar to those of the R647 are achieved at a higher anode to cathode supply voltage, namely about 1250 volts. Since the R1635 has only eight dynode stages, while the R647 has ten, the R1635 requires a higher anode to cathode voltage than the R647 in order to obtain a similar response. Moreover, the R1635 is approximately 50% more expensive than the R647. Because of budget and time constraints we were unable to obtain any of the R1635 for the current phase of the Aerosol project. If APDs should prove to be unsuitable, these miniature PMTs might be preferable to the R647 type in situations where space is at a premium. However, other factors, such as the need for greater power, might offset the advantage of some size reduction.

In the light of the failure of other detection systems to match the required characteristics of PMTs, it was decided to continue to use PMTs in the Phase II instrument. However, avalanche photodiodes should be explored further in a future phase. Miniature PMTs might be suitable if APDs cannot be used.

2.4 *Electronics.*

The aerosol system includes four types of electronic circuits. (1) The photomultiplier tube amplifier boards consist of two-stage linear amplifiers which provide the necessary range for amplifying the PMT output currents without the unacceptably slow response time of logarithmic amplifiers. (2) The peak/threshold detector monitors a software selectable trigger and reference channel. Upon receiving a signal above the user designated threshold level, the circuit tracks the particle signal until a peak is detected. It then saves all the scattered intensities. (3) The control board coordinates the operation of the other boards based on user-supplied instructions received from the system computer. The control board also sends data to the computer. (4) The computer/instrument interface consists of a Data Translation 2821 analog to digital board. Appendix A describes its operation in detail.

2.5 *Data Processing Software.*

The preprocessor program developed for data collection from the Aerosol Particle Analyzer functions in the following way.

A trigger channel is selected by the user and a threshold signal level is entered. At the same time a reference channel is chosen with its corresponding threshold value. These two channels may be selected from any of the 24 available, which can, of course, be at any of the available angular locations. When the data collection routine, AEROSOL, is activated, the A/D channel corresponding to the trigger is constantly monitored by the computer. If the signal from this channel exceeds the threshold level, the reference channel signal on the A/D board is read to see if it too is above its threshold. If both signals are above their respective threshold levels, then the signal on the trigger channel is monitored for a peak value, at which point the signals at all channels are held by the sample and hold circuits. These signals are then read by the computer from both stages of the dual amplifiers. Subsequently, the Analyze Data Module segment of the software selects the largest non-saturating signal from each amplifier pair, subtracts previously measured and stored background offsets, and finally multiplies by a normalization factor to compensate for differences between individual detectors. The resulting 24 numbers are stored on the hard disk.

Integration of the scattered signal over the particle's entire path through the laser was considered as a means for improving the signal quality, but this would distort the measurements for many classes such as irregular, tumbling particles. Accordingly, we let the peak signal detected at one channel trigger the capture and conversion of signals at the other angles. To this end, it was necessary to eliminate spurious trigger events caused by noise. This was achieved by using the trigger signal on *two* channels as described above, which also minimized the delay time between peak detection and sample-and-hold (see Appendix B). Collected data can be displayed numerically or graphically and plotted in either the θ or ϕ planes.

Real time particle characterization using the Definicon board and the strip map technique[3] was investigated and is expected to be completed and implemented by Company funding during the next year.

2.6 *Aerosol Handling Hardware.*

A set of pumps, valves and regulators sample, dilute and introduce aerosol particles entrained in a fine laminar stream through the center of the chamber intersecting the laser beam, one particle at a time. To achieve this, air flow of both the input and output of the scattering chamber is controlled by pumps and valve systems. Furthermore, the input can be made up of three components. First, there is a sample stream: this can be sent directly through the chamber or it may be diluted with 0.2 μm filtered air in order to dilute the particles and ensure that only one be in the beam at a given time. Finally, a filtered air sheath flows around the particle stream in order to prevent turbulence at the interface with the static air in the chamber.

3. CONCLUSIONS

The aerosol system incorporating the final instrument design was delivered in December 1987 and preliminary data are impressive. Results from the first system have recently been published as the cover feature of *Applied Optics* (January 15, 1988) and we expect results from the second instrument to be even more significant. We believe that the instrument design will prove its great potential in aerosol characterization in the years to come. Furthermore, we feel that the two avenues we explored but never implemented, that is, solid state detectors and real-time particle characterization, are tremendously promising and we intend to implement them during the commercial exploitation of the system. The system was offered commercially in February 1988 and the first editorial follow up appeared in various trade journals in late March. The first systems to be offered commercially will have limited collection and analytical software. It is expected that these systems will be for research purposes at large industrial laboratories. The company has developed a strategy for the implementation of the strip map technique, but will require a large data base to test this approach. These data will be obtained from cooperative software development contracts with early users of the system.

LITERATURE CITED

1. Philip J. Wyatt and Steven D. Phillips, US Patent #4693602, "Method and apparatus for measuring the light scattering properties of small particles", issued 15 September 1987.

2. Philip J. Wyatt, Kevin L. Schehrer, Steven D. Phillips, Christian Jackson, Yu-Jain Chang, Randall G. Parker, David T. Phillips, and Jerold R. Bottiger, "Aerosol Particle Analyzer", *Applied Optics* **27**, 217-221 (1988).

3. Gregory M. Quist and Philip J. Wyatt,"Empirical solution to the inverse-scattering problem by the optical strip-map technique", *J.Opt.Soc.Am.*(A) **2**, 1979-1985 (1985).

Blank

APPENDIX A

SUB-MICRON PARTICLE ANALYZER

ELECTRONICS: THEORY OF OPERATION

The electronics for the Aerosol system comprises three distinct types of circuit board, each type being designed with modularity and future expansion in mind. The three types are: 1) Photomultiplier (PMT) amplifier board; 2) peak detector board; 3) control board.

Each PMT amplifier board inputs the PMT signal and amplifies it to levels suitable for input to the rest of the electronics. The peak detector board monitors a user-selected channel for the occurrence of a maximum or "peak" signal, and sets a gate when such a peak occurs. The control board receives instructions from the computer and, based on these instructions, controls the operation of the other two types of board. For the most part, these computer instructions are easily understood through the prompts provided the user, and the system is easy to operate. Figure A1 shows a block diagram of the electronics system.

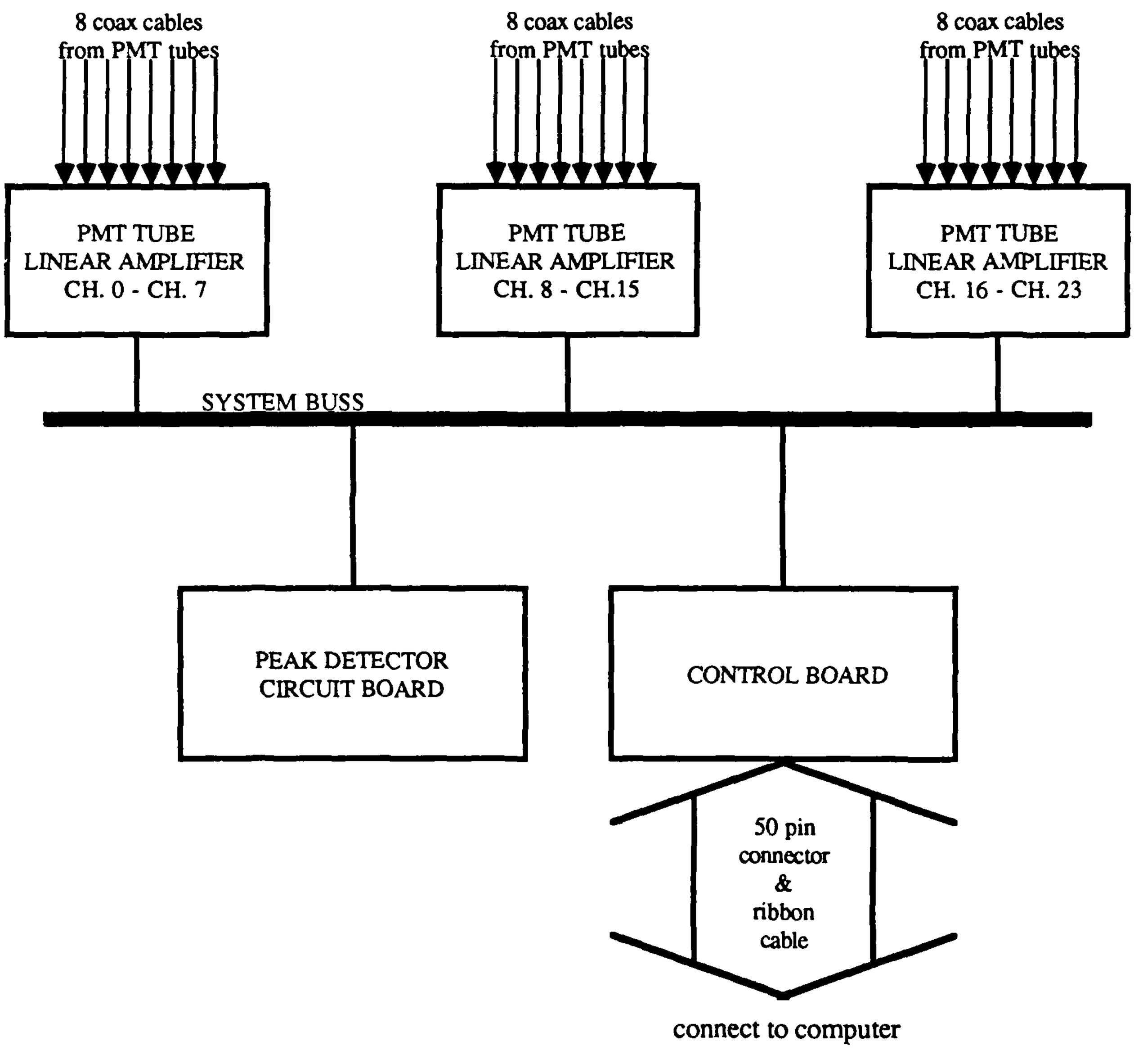

Figure A-1. Aerosol Electronics System

1. *PMT Amplifier Boards.*

Each PMT amplifier board has eight channels, *i.e.* we need one board for every eight PMTs. The output current of each PMT ranges from about 50 nanoamps to about 200 microamps. We obtain the necessary dynamic range by using a two-stage linear amplifier. Each stage uses the OP-27 operational amplifier.

The first stage converts the current output of the PMT into a voltage, with a transconductance of 50 millivolts per microamp. The second stage is a gain 100 voltage amplifier.

2. *Peak/Threshold Detector Board.*

Some of the amplifier channels have been pre-selected for use in particle detection. The outputs from these channels are relayed to the peak detector circuit. When a particle passes through the laser beam, a signal and peak results. A valid peak must be at least a user-specified amount above the background signal, which is obtained while the laser is on with no particle present in the beam. When a valid peak signal is transmitted to the peak detector, the peak detector ends a hold signal to the sample/hold circuit. The sample/hold circuit receives a signal from both stages of each amplifier; when the hold signal is received, these amplified signals are sent to the DT2821 A/D board, which is plugged into one of the bus connectors inside the computer. This board converts the analog signals into digital form. The software handles the storage and processing of the digitized data. The DT2821 converts 16 channels at a time. There are a total of 48 signals held and converted in this manner.

One problem concerns discrimination of noise, which might seem to be a peak. To prevent this, we select *two* detectors or channels to use in determining when a particle has passed through the beam. A peak must occur within about 20 μs on *both* the trigger and reference channels in order for a "hold data" signal to be activated. If a peak occurs on only one of these channels, we assume this to be PMT noise and do not process these data further.

3. *Control Board.*

Commands to the control board come from the computer and are transmitted to the digital output ports of the DT2821. These commands are easily understood from computer prompts provided the user. They are generated by the software in order to control data acquisition. Data is collected in Direct Memory Access (DMA) mode for high speed operation. Before the start of data collection, the control board sends a command to reset the peak detectors. As soon as a peak is detected, the peak/threshold detector board sends a trigger command to the control board. The control board then sets the Sample/Hold (S/H) command line to high. As described above, these data are digitized and stored in the computer for later processing.

Blank

APPENDIX B

SOFTWARE MANUAL

1. AEROSOL - DATA COLLECTION PROGRAM.

This program records the user-selected detector placement and PMT configuration for the submicron Aerosol Particle Analyzer (APA). In addition, it directs the measurement of the PMT dark offsets, background offsets and detector calibration coefficients. It also allows on-screen or printer display of the unsorted data.

The program is loaded by typing AEROSOL at the DOS prompt. The main menu will appear on the screen.

The main menu allows the user to select each of the following options by typing the corresponding number on the menu then pressing return.

1 Measure Dark Offsets

2 Measure Background

3 Calibration

4 Collect Data

5 Analyze Data

6 Print/Edit Master Table

7 Exit Program

Before the program is started, the system configuration must be entered in the Master Table (item 6). This consists of the angular positions of the 24 detectors, the alignment of the polarizer for each detector, and the PMT voltage of each detector channel. The dark offsets, background offsets, and calibration coefficients will be entered automatically in this table as they are measured (items 1, 2, 3). These values will be saved on the disk and need only be changed (item 6) when the detector configuration is changed.

At the start of each day's measurements, the dark and background offsets should be measured. The dark offsets are a measure of any dark current from the PMTs and electronics, as well as an indication of any light leaks that may be present in the read head. The background offsets are a measure of the scattering from the atmosphere in the read head, as well as stray light effects.

The calibration coefficients compensate for differing sensitivities among detector channels. The signal from a standard light source is used to calculate the coefficient for each detector.

Item 4 initiates data collection and 5 normalizes these data to a specified channel, as well as permitting the display of signal distributions by channel or particle.

1.1 *Measure Dark Offsets.*

When this option is selected, the operator will be prompted to switch off the laser. The dark offsets will then be measured repeatedly and displayed on the screen. Press <esc> to save these values and return to the main menu. The values are saved in the OFFSET.TBL file.

1.2 *Measure Background.*

When this option is selected, the operator will be prompted to turn the laser on. The background offset will be measured repeatedly and displayed on the screen.Press <esc> to save these values and return to the main menu. The values are saved in the BACK.TBL file.

1.3 *Calibration.*

Before calibrating measure the dark offsets (menu item 1). The background offsets are ignored in this measurement. First, select a reference channel to which all the other detectors will be calibrated. The calibration coefficient for this channel is set at 1.0. Then, one at-a-time, each detector from 0 to 23, should be placed as prompted so that it receives a constant amount of light, and the signal measured. The calibration coefficients are then calculated automatically.

1.4 *Collect Data.*

Data are collected as follows. The user selects a trigger and a reference channel, with a threshold value for each. When the light intensities measured exceed the corresponding threshold values on both of these channels, the sample and hold circuitry starts looking for a peak, i.e., a *decrease* in scattered intensity on the trigger channel. As soon as this signal starts to decline, all 24 channels are read and the intensities stored in the PEAK.TBL file.

After selecting item 4, select a trigger *and* a reference channel from the available lists by moving the cursor up and down. Then enter a threshold for each in millivolts. This is the total signal received by the detector. The value entered should be greater than the background scattering level.

The program will then start looking for peak values above these thresholds, and will display them on the screen. (Because data can be collected faster than it can be shown on the screen not every particle is displayed.) If no signals above threshold are found, the top of the screen will remain blank. The threshold values can be altered at this point: + or - keys increase or decrease the trigger threshold by 10 millivolts, and H and L keys do the same for the reference channel threshold. Type <esc>, and the peak values will start being saved to disk. The number of particles saved, in groups of 32 will be displayed on the screen. Type <esc> once more to stop collection and return to the main menu.

1.5 *Analyze Data.*

This section normalizes the data collected to the reference channel specified during calibration, and saves these data in a file. It is then possible to view the distribution of the intensities for a given channel and to view the data for a given particle plotted against channel number. Prompts are easily understood to achieve the functions.

1.5.1 *Normalize Data.*

This option takes the data just collected (which is stored in the PEAK.TBL file) and writes them to a user specified file in normalized and, finally, ASCII form. For each particle, the normalization channel, chosen by the user, is set to a value of 1 and the others are normalized according to the values generated in the calibration mode (3).

1.5.2 *View Each Channel Distribution.*

Select a channel, and the distribution of intensities over the particles collected is displayed on the screen.

1.6 *Print/Edit Master Table.*

The master table contains the following information for each of the 24 detectors: the angular position in the read-head defined in terms of the scattering angles θ and ϕ, the alignment of the polarizer on its corresponding detector, the photomultiplier tube voltage, the dark and background offsets, and the calibration coefficients. When the dark offsets, background offsets or calibration coefficients are measured, they are automatically entered in the table. The polarization, angular position, and PMT voltage for each detector must be entered manually by moving the cursor to the correct position on the table and typing the entry then pressing return. The table covers two screens; the first displays detectors 0 - 11, the second 12 - 23. Typing "K" moves between them. Type <esc> to save the entered values and return to the main menu.

2. VIEWDATA - DATA GRAPHING PROGRAM

This program allows you to plot the data from a specified file particle by particle against either θ or ϕ. Type VWDATA at the DOS prompt.

2.1 *Enter Filename.*

The filename for the data to be viewed should have a .INT suffix, files processed in AEROSOL automatically have this.

The data can then be plotted in two ways,

2.2 *Plot Data vs. Phi*

2.3 *Plot Data vs. Theta*

Data may be plotted against θ or ϕ. When the data are loaded in, the program reads the angular positions of the detectors from the Aerosol master table and sorts the channels into groups of equal θ, or equal ϕ. Then, for example, to plot data against ϕ, after selecting option 2, select which group of ϕ values to plot from the ones displayed on the screen, and then select the particle number. The group of channels for the chosen particle, will be plotted on the screen (Fig. B1).

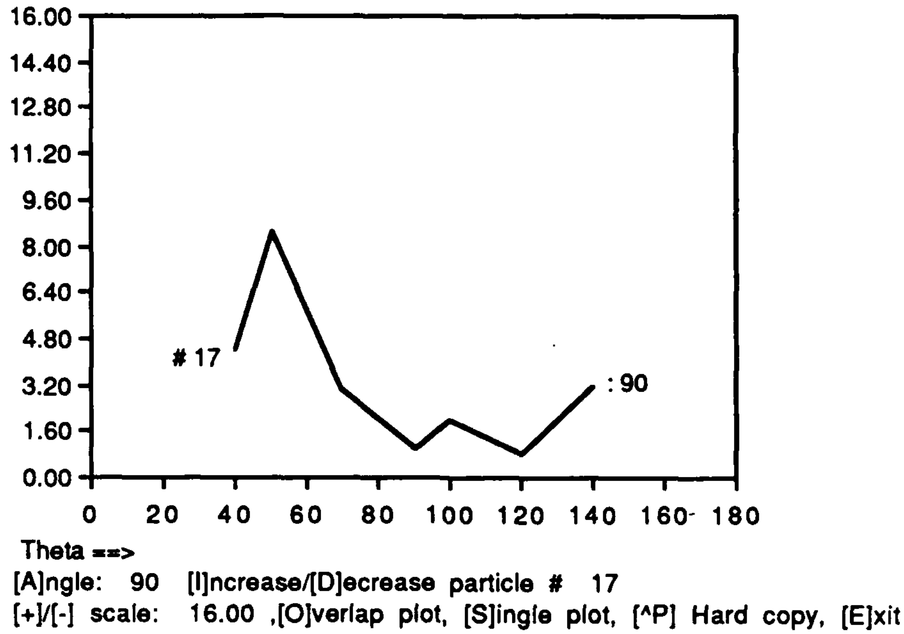

Figure B-1 Viewdata Display

It is then possible to change the display in the following manner:

Typing "A" increments the angular group to be plotted to the next one.

Typing "I" or "D" respectively increases or decreases the particle number to be plotted by one.

Typing "+" or "-" respectively increases or decreases the intensity scale of the plot (1 is equal to the intensity of the normalization channel). It is also possible to select a logarithmic scale by selecting "l", the scale option shown on the screen before 0.25.

Typing "O" overlays the chosen plot with the current display, while "S" clears the current display and just shows the chosen plot.

Typing "Control-P" prints out the screen.

Typing "E" returns to the Main Menu.

Blank

APPENDIX C

PATENTS

The following U.S. Patents were developed during this contract and its earlier phases:

1. US Pat. No. 4548500
   Process and apparatus for identifying and characterizing small particles.
   Philip J. Wyatt and Gregory M. Quist
   Oct. 22, 1985

2. US Pat. No. 4693602
   Method and apparatus for measuring properties of small particles.
   Philip J. Wyatt and Steven D. Phillips
   Sept. 15, 1987

3. US Pat. No. 4710025
   Process for characterizing suspensions of small particles.
   Philip J. Wyatt and Gregory M. Quist
   Dec. 1, 1987